

# RESERVE COPY PATENT SPECIFICATION

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## COMPLETE SPECIFICATION.

### Improvements in and relating to the Production of Phosphorus-halogen and Phosphorus-halogen-oxygen Compounds.



We, N. V. ELECTROCHEMISCHE INDUSTRIE, of Molenweg 20, Roermond, Holland, a Dutch Company, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

The present invention relates to the production of phosphorus-halogen and phosphorus-halogen-oxygen compounds.

In our prior Application No. 302,927 a general method for the preparation of inorganic halides was described which consisted in subjecting a heated mixture of an oxygenated compound and carbon to the action of halogen. It was suggested that such a reaction might be accelerated by chemical, physical or mechanical means such, for instance, as by the action of actinic rays, catalysts, stirring and the like. In an example of this prior Application calcium triphosphate admixed with excess of carbon was treated with chlorine at 600° C.

According to the present invention the reaction which occurs at raised temperature between oxygenated phosphorus compounds and halogen in the presence of carbon may be considerably accelerated by carrying it out in the presence of substances belonging to one of the following two groups:

1. Materials such as molten zinc chloride and molten sodium carbonate, in which the reacting substances are more or less soluble or become finely divided and which accordingly act as dispersing agents for the reacting materials.

2. Metal chlorides such as ferric chloride and copper chloride.

It has already been proposed to prepare chlorides by subjecting an oxide to the action of chlorine in the presence of carbon and in suspension in a fused chloride or mixture of chlorides. It has also been suggested in the preparation of chlorides from oxides to subject the oxides in the presence of carbon to the action of chlorine associated with sulphur chloride for the purpose of facilitating the reaction.

The present invention, however, is concerned with a process for the production

of phosphorus-halogen compounds from phosphates and carbon in connection with which it has been found that the accelerating agents above mentioned produce considerable improvement in the reaction.

The process is of special importance with the reaction of tricalcium triphosphate with chlorine in the presence of carbon, in which phosphorus-chlorine compounds as well as  $\text{CaCl}_2$  are formed. The process is further explained by the following examples of execution:

#### EXAMPLE I.

A mixture of tricalcium phosphate and carbon in excess is dried and mixed with 5% of zinc-chloride. This mixture is heated to 500—600° C. while a stream of chlorine is conducted over it. The reaction takes place easily, phosphorus-chlorine compounds being formed, which distil over with the carbon monoxide also produced, while calcium chloride remains as residue.

#### EXAMPLE II.

To a dry mixture of tricalcium phosphate with carbon in excess, 2% of cupric chloride is added. In the same way as described in the first example, chlorine is conducted through the mixture whereby the production of phosphorus-chlorine compounds and calcium chloride takes place, giving a very good yield.

The reaction may further be accelerated by physical and mechanical influences, for example, by agitation of the mixture.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. A process for the production of phosphorus halides and phosphorus oxyhalides from phosphates, in which phosphates mixed with carbon are subjected to the action of halogens in the presence of one or more of the materials hereinbefore specified for the purpose of facilitating the reaction.

2. A process as claimed in claim 1, in which the phosphorus compound treated is tricalcium phosphate.

3. A process as claimed in claim 1 or 2, in which zinc chloride is employed.

[Price 1/-]

4. The process for the production of phosphorus-halogen compounds substantially as described.

5. Phosphorus-halogen compounds when produced by the process claimed in any of the preceding claims.

Dated this 30th day of May. 1930.

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